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(54) Title: RUBBER BASED HOT MELT ADHESIVES WITH IMPROVED WICKING PROPERTIES WITH LOW LEVELS OF SURFACTANT

(57) Abstract: A water sensitive or water activated hot melt adhesive composition comprising: (a) 5 to 40 % by weight, of an A-B-A linear or radial rubbery block copolymer; (b) 20 to 70 % by weight of a compatible tackifying resin; (c) 0 to 30 % plasticizer; (d) 0 to 2 % stabilizer; and (e) one or more of the following; (i) 0.1 to 20 % of one or more polyethers; (ii) 0.1 to 9 % of a surfactant; or (iii) a combination of (i) and (ii). The adhesive composition exhibiting a contact angle of 90° or less and a reduction in surface tension of less than about 40 dynes/cm. The present invention is also directed to a disposable article containing an absorbent core bound together or reinforced with a hot melt adhesive.

RUBBER BASED HOT MELT ADHESIVES WITH IMPROVED WICKING PROPERTIES WITH LOW LEVELS OF SURFACTANT

FIELD OF THE INVENTION

This invention relates to hot melt adhesives for bonding nonwoven fabrics and tissues, and labeling applications.

BACKGROUND OF THE INVENTION

A nonwoven fabric is an interlocking network of synthetic or naturally occurring fibers, or a combination of the two, in which the individual fibers are mechanically, chemically, or thermally bonded to each other. Tissue is a closely related material in which the individual fibers may or may not be bonded to one another. The fabric or tissue is characterized by flexibility, porosity and integrity. Nonwovens are used commercially for a variety of applications including insulation, packaging, household wipes, surgical drapes, medical dressings, and disposable articles, such as, diapers, adult incontinent products and sanitary napkins.

In many of the end use applications it is necessary to adhere the nonwoven or tissue to another substrate or component. The second substrate may be another nonwoven, tissue, or an unrelated material, such as a polyethylene film. Commonly, hot melt adhesives are employed to bond the assembly together. Hot melt adhesives allow for cost and time efficient manufacturing since there is no evaporation step necessary as is the case for water based or solvent based adhesive systems. For nonwoven applications, suitable hot melt adhesives must possess good flexibility (or hand); no staining or bleed through; suitable viscosity, set speed and open time to function on

commercially available equipment; and finally, acceptable thermal aging properties.

Rubbery block polymers are often used for these constructions; however, these block polymers are not generally water soluble/sensitive. For various applications, it is also desired that some hot melt adhesives be hydrophilic, i.e., be water-sensitive or water-activated. Such hydrophilic adhesives find use, for example, in the construction of flushable disposable products including diapers and sanitary napkins where the high degree of tack which is needed during construction and use must be substantially decreased so as to prevent adhesion to porcelain and sewer pipes. Hydrophilic adhesives are also useful in certain applications where easy-clean up of the application equipment is desirable.

Rubber-based adhesives with low water contact angle have been formulated using high levels (10-50%) of non-ionic surfactants (Kauffman U.S. Patent No. 5,532,306). These formulations also employ very polar resins, such as rosin, rosin esters, and terpene phenolics, with partially saponified rosin being most preferred. While these resins are suitable for many applications, in some applications such as personal care products (eg. diapers and sanitary napkins) the odor of these more polar resins can be unacceptable.

Further, high levels of surfactant are undesirable as they 1) present more risk to the consumer of skin irritation, and 2) will generally have a larger negative effect on the surface tension of the fluid being absorbed by the personal care article. If the fluid's surface tension is reduced through contact with the adhesive, the rate at which it will subsequently wick deeper into the

article is retarded (C. Nederveen, TAPPI Journal, 77 (12), p.174.), having a negative impact on the article's overall effectiveness at rapid fluid absorption.

Fluorochemical surfactants have been shown to be effective at levels as low as 2.5% in rubber-based formulas (Raykovitz U.S. Patent No. 5,804,519). However, these surfactants suffer from the disadvantages of very high cost, and the capacity to be absorbed through the skin and accumulate in the body with potential adverse health effects, based on animal studies.

Olefin copolymer -based adhesives formulas which use low levels of surfactant have been disclosed (Paul U.S. Patent No. 5,685,758). However, olefin-based adhesives lack the high level of pressure sensitivity and flexibility obtainable with rubber-based products. In addition, olefin-based adhesives containing surfactant do not release from the substrate when exposed to moisture as many rubber-based adhesives do. This feature is desirable in applications such as flushable sanitary napkins and recylable bottles. For flushable sanitary napkins it is desirable that the positioning adhesive lose its tack and the construction adhesive allow the various layers to debond once the napkin is flushed. When recyIcling bottles it is desriable for the adhesive holding the label to release from the bottle when exposed to tap water, thus permitting easy seperation of the label from the bottle.

Thus, a need exists for rubber-based adhesives which exhibit water sensitive attributes (low contact angle and/or releaseability), but with low odor, low cost, acceptable health and safety attributes, and minimal surface tension reduction.

SUMMARY OF THE INVENTION

The present invention is directed to water sensitive or water acitvated rubber based hot melt adhesives.

Specifically, the present invention is directed to a hot melt adhesive composition comprising:

(a) 5 to 40% by weight, of an A-B-A linear or radial rubbery block copolymer;

(b) 20 to 70% by weight of a compatible tackifying resin;

(c) 0 to 30% plasticizer;

(d) 0 to 2% stabilizer; and

(e) one or more of the following:

 (i) 0.1 to 20% of one or more polyethers;

 (ii) 0.1 to 9% of a surfactant; or

 (iii) a combination of (i) and (ii).

The surfactant will cause the adhesive composition to exhibit a contact angle of 90° or less and a reduction in surface tension of less than about 35 to 40 dynes/cm, preferably less than or equal to 35 dynes/cm, preferably 30 dynes/cm, more preferably 20 dynes/cm, most preferably 15 dynes/cm.

DETAILED DESCRIPTION OF THE INVENTION

The rubbery (elastomeric) component of the compositions of the present invention are linear or radial block copolymers having the general configuration: A-B-A

wherein the polymer blocks A are non-elastomeric polymer blocks which, as homopolymers have glass transition temperatures above 50°C, while the center

elastomeric polymer blocks are derived from at least one conjugated diene such as butadiene or isoprene. These mid-blocks may, if desired, be partially or substantially hydrogenated. Further, they may be linear or branched. Typical branched structures contain a mid-block portion with at least three branches which can radiate out from a central hub or can be otherwise coupled together.

While the specific molecular weight of the block copolymer prepared from the conjugated diene and the non-elastomeric terminal blocks may be varied for specific end uses, it is preferred that the elastomeric center blocks have an average molecular weight from about 15,000 to about 250,000, preferably 24,000 to 150,000, and that they comprise from 50 to 90% by weight of the entire block copolymer. The terminal blocks which comprise the remaining 10 to 50% of the copolymer are those having a number average molecular weights between 5,000 and 125,000, preferably 5,000 to 25,000. These terminal blocks are prepared by polymerization of vinyl aromatic monomers and should have glass transition temperature above about 50°C, and the difference in glass transition temperature between that of the center block and of the end blocks should be greater than about 100°C.

The non-elastomeric terminal blocks preferably comprise homopolymers or copolymers of mono vinyl aromatic monomers such as styrene, vinyl toluene, vinyl xylene, ethyl vinyl benzene as well as bicyclic monovinyl compounds such as vinyl naphthalene and the like.

The center elastomeric blocks are prepared from conjugated dienes such as isoprene, butadiene, copolymers of styrene and butadiene as well as their homologues. Additionally, these elastomeric blocks may be partially or substantially hydrogenated.

The non-functionalized block copolymers may be prepared using methods familiar to one of skill in the art, or they may be obtained from manufacturers such as Shell Chemical Company, Dow Chemical, Fina Ltd. in the Netherlands, or Firestone.

Preferably, the block copolymers useful herein are comprised of styrene and butadiene blocks arranged in a substantially radial configuration and containing least 35%, and generally up to about 50%, by weight of the styrene moiety. Most preferred for use herein is STEREON 841A, an SBS copolymer comprising 43% styrene from Firestone.

The hot melt adhesive of the present invention also comprises 20 to 70% by weight of a compatible tackifying resin. The tackifying resins which are preferably used in the adhesive compositions must be compatible with the polymers and are generally polar in nature and have a Ring and Ball softening point greater than 60°C. More particularly, the useful tackifying resins include any compatible resins or mixtures thereof such as (1) natural and modified rosins such, for example, as gum rosin, wood rosin, tall oil rosin, distilled rosin, hydrogenated rosin, dimerized rosin, maleated rosin and polymerized rosin; (2) terpene resins, (3) phenolic modified terpene resins and hydrogenated derivatives thereof such, for example, as the resin product resulting from the condensation, in an acidic medium of a bicyclic terpene and a phenol and (4) C_5, C_5/C_9, C_9 and C_{10} aliphatic or aromatic tackifying resins. Mixtures of two or more of the above described tackifying resins, as well as blends of the above resins with small amounts of (e.g., less than about 10% of the adhesive) less compatible resins may be utilized for some formulations. For water releasable applications, the use of rosin based tackifiers such as

FORAL NC, a hydrogenated rosin available from Hercules Incorporated, is preferred. If present, the tackifier may comprise up to about 70% of the adhesive; however, it is generally used in amounts of about 20 to 60% by weight.

Most preferred for use herein is a C_5/C_9 hydrocarbon tackifier have a Ring and Ball softening point of 103°C such as ECR 179EX from Exxon Chemical.

Various plasticizing or extending oils may also be present in the composition in amounts of up to about 30%, preferably 0 to 25%, by weight in order to provide wetting action and/or viscosity control. The above broadly includes not only the usual plasticizing oils, such as mineral oil which is preferred, but also olefin oligomers and low molecular weight polymers, as well as vegetable and animal oils and their derivatives. Petroleum derived oils that may be employed are relatively high boiling materials containing only a minor proportion of aromatic hydrocarbons (preferably less than 30% and, more particularly, less than 15% by weight of the oil). Alternatively, the oil may be totally non-aromatic. The oligomers may be polypropylenes, polybutenes, hydrogenated polyisoprene, hydrogenated polybutadiene, or the like, having average molecular weights between about 350 and about 10,000. Vegetable and animal oils include glyceryl esters of the usual fatty acids and polymerization products thereof.

Also useful as plasticizers are polar synthetic compounds, such as the aliphatic and aromatic polyester plasticizers available from C.P. Hall Co., Stow, OH. Amides phosphate esters, sulfonamides, and phthalates are also suitable at varying levels.

An antioxidant or stabilizer may also be included in the adhesive compositions in amounts of up to about 2% by weight. Among the applicable antioxidants or stabilizers are high molecular weight hindered phenols and multifunctional phenols, such as sulfur and phosphorous-containing phenols. Representative hindered phenols include: 1,3,5-trimethyl 2,4,6-tris (3,5-di-tert-butyl-4-hydroxy-benzyl)benzene; pentaerythritol tetrakis-3(3,5-di-tert-butyl-4-hydroxyphenyl)-propionate; n-octadecyl-3,5-di-tert-butyl-4-hydroxyphenol)-propionate; 4,4'-methylenebis (2,6-tert-butylphenol); 4,4'-thiobis (6-tert-butyl-o-cresol); 2,6-di-tertbutylphenol; 6-(4-hydroxyphenoxy)-2,4-bis(n-octyl-thio)-1,3,5-triazine; di-n-octadecyl 3,5-di-tert-butyl-4-hydroxy-benzyl-phosphonate; 2-(n-octylthio)-ethyl 3,5-di-tert-butyl-4-hydroxy-benzoate; and sorbitol hexa[3-(3,5-di-tert-butyl-4-hydroxyphenyl)-propionate].

In addition to the above, the hot melt adhesives of the present invention will also comprise one or more polyethers and/or a surfactant. Therefore, in one embodiment of the present invention, the adhesive comprises

(a) 5 to 40% by weight, of an A-B-A linear or radial rubbery block copolymer;

(b) 20 to 70% by weight of a compatible tackifying resin;

(c) 0 to 30% plasticizer;

(d) 0 to 2% stabilizer;

(e) 0.1 to 20% of one or more polyethers; and

(f) 0.1 to 9% of a surfactant.

In another embodiment, the hot melt adhesive of the present invention comprises:

(a) 5 to 40% by weight, of an A-B-A linear or radial rubbery block copolymer;

(b) 20 to 70% by weight of a compatible tackifying resin;

(c) 0 to 30% plasticizer;

(d) 0 to 2% stabilizer; and

(e) 0.1 to 9% of a surfactant.

In a further embodiment, the hot melt adhesive comprises:

(a) 5 to 40% by weight, of an A-B-A linear or radial rubbery block copolymer;

(b) 20 to 70% by weight of a compatible tackifying resin;

(c) 0 to 30% plasticizer;

(d) 0 to 2% stabilizer; and

(e) 0.1 to 20% of one or more polyethers.

If present, the surfactants will be used in amounts of 0.1 to 9%, preferably 0.1 to 1% by weight, but the exact preferred range will depend on the individual adhesive system. It has been found that in the adhesives of the present invention, less surfactant is needed than with other rubber based systems.

Suitable surfactants include nonionic and anionic surfactants. Exemplary nonionic surfactants are:

ethoxylates of (i) C_1-C_{18}, preferred C_8-C_9 alkyl or dialkyl phenols, such as those sold under the tradenames MACOL DNP-10, available from PPG Industries, Gurnee, Illinois, a 10 mole ethoxylate of dinonyl phenol, and TRITON X-100, available from Union Carbide, a 10 mole ethoxylate of octyl phenol; (ii)

alkyl C_8-C_{60} mono-alcohols, such as those sold under the tradenames SURFONIC L-12-8, an 8 mole ethoxylate of dodecanol, available from Huntsman Chemical Co., and UNITHOX 480, a 38 mole ethoxylate crystalline surfactant available from Petrolite Specialty Polymers Group, Tulsa, OK; and (iii) propylene oxide polymers, such as those sold under the tradename PLURONIC, which are ethylene oxide/propylene oxide block copolymers having a Mn of 200 to 3000 available from BASF; and

benzoates formed by partial condensation of benzoic acid with hydrophilic di or mono-ols having less than 1000 Mn, such as the product of condensing about three equivalents of benzoic acid with four equivalent of diethylene glycol, commercially available as XP 1010 from Velsicol Chemical.

The preferred nonionic is GENAPOL a linear alcohol ethoxylate from Clariant.

Suitable anionic surfactants are:

C_8-C_{60} alkyl ethoxylate sulfonates, $(CH_3\text{-}(CH_2)_{11\text{-}14}\text{-}(O\text{-}CH_2CH_2)_3\text{-}SO_3^{-}Na^{+}$, such as, AVENEL S30, available from PPG Industries;

dialkyl C_4-C_{60} sulfosuccinates, such as di(2-ethylhexyl)sulfosuccinate available from Cytec Inc. under the tradename AEROSOL 0T-100;

alkyl C_8-C_{60} sulfonates, such as, RHODAPON UB ($C_{12}\text{-}SO_3^{-}Na^{+}$) available from Rhone Poulenc; and

alkyl/aromatic sulfonates.

The most preferred anionic surfactant is AERSOL OT 100, a di(2-ethylhexyl)-sulfosuccinate available from Cytec Inc. Overall, the anionic surfactants are preferred in the present adhesives.

Examples of suitable silicone surfactants include, but are not limited to,

ethoxylates or propoxylates of polydimethyl siloxane, having a number average molecular weight of 500 to 10,000, preferably 600 to 6000, such as are sold under the tradenames SILWET L-77, L-7605, L-7500, and NUWET 550 available from OSi Specialties, Danbury, Connecticut; and product 193 from Dow Corning.

The preferred surfactants are those with lower molecular weights because these have increased compatibility in the adhesive formulations. The maximum acceptable molecular weight depends on the type of surfactant and the other ingredients in the adhesive formulation.

The hot melt adhesive composition of the present invention may also comprise up to 20% of one or more polyethers. The polyether will assist the surfactant in lowering the contact angle. Therefore, by using polyehters, less surfactant can be used. Sources for polyethers for use in the subject hot melt adhesive compositions are known to those of ordinary skill in the art. For example, polypropyleneglycol may be obtained from Arco or Bayer. Polyethers may also be synthesized using any method known in the art.

The polyethers of this invention consist essentially of homo, alternating, or random -R-O- units. For this invention "consisting essentially of" means that there may be one or more units in a polyether which is not a -R-O- as defined above, so long as the properties of such a polyether are not significantly changed. The term "homo" means that the R of each -R-O- unit is the same for all -R-O- units in the polymer. The term "alternating" means that the polyether comprises two or more different alkyl groups for R, and that the -R-O- units having each different type alternate with one another. In one embodiment, the polyether comprises two different types of units which alternate. In another

embodiment, the polyether comprises three different types of units which alternate with one another. The term "random" means that the polyether has two or more different alkyl groups for R, and that the units containing these different R groups are randomly dispersed in no predictable pattern within the polymer.

The alkyl moieties for R comprise 2 or more carbon atoms. Preferably, the alkyl moieties have from 2 to 5 carbon atoms, for example 3 carbon atoms or 4 carbon atoms. The alkyl moieties for R may be saturated or unsaturated, or cyclic or acyclic. R may be straight or branched, aromatic or aliphatic. Examples of polyethers which may be used for the subject invention include, but are not limited to, PPG and polytetrahydrofuran. The subject invention may comprise a single polyether (i.e., a "mixture" of one polyether) or a combination of polyethers (i.e., a "mixture" or more than one polyethers).

Polyethers useful for the subject invention are of various molecular weights. Preferably, polyethers which are used for the invention have a molecular weight of 425 Daltons or higher, a molecular weight of between about 2000 Daltons and about 4000 Daltons being more preferred.

Preferred polyethers are polypropylene glycol, PPG, and polytetrahydrofuran.

If PPG is used as a polyether for this invention, the PPG preferably has a molecular weight between about 2000 and about 4000 Daltons. In one embodiment, the PPG of the subject hot melt adhesive composition has a molecular weight of about 3000. Preferred is PPG 3025, a polypropylene glycol available from Arco with molecular weight of about 3000.

The amount of polyether used in the subject adhesive compositions is

that amount which will cause a fluid contact angle of less than about 90 and a fluid surface tension reduction of less than about 40 dyne/cm, and one of ordinary skill in the art can determine a suitable amount of polyether accordingly, by testing the fluid contact angle and surface tension reduction of a composition comprising polyether using known techniques. In one embodiment the fluid contact angle is less than about 70 degrees, more particularly less than about 50 degrees. In a further embodiment the fluid contact angle is less than about 25 degrees.

In another embodiment, the polyether is added in an amount such that the surface tension reduction is less than about 30 dyne/cm, most preferably less than about 20 dyne/cm.

In addition to the above, polyether/surfactant blends such as NUWET 500, NUWET 500, a hydrophilic blend of >65% organomodified polydimethyl siloxane, <20% polyalkylene oxide, and <20% ethoxylated alkyl or NUWET 550, a polyalkyleneoxide-modified polydimethylsiloxane both available from Osi Specialties may be used.

It has been found, in accordance with the present invention, that combining a surfactant with polyethers as defined above in a hot melt adhesive composition further improves the repeat strike through time and fluid wicking abilities of absorbent articles comprising such adhesive compositions. Thus, in one embodiment, the present invention is directed to an absorbent article comprising the rubber based hot melt adhesives described above.

Optional additives may be incorporated into the hot melt compositions in order to modify certain properties thereof. Among these additives may be included colorants such as titanium dioxide; synthetic or natural waxes; and

fillers such as talc, etc.; wetness indicators such as Basacid Blue moisture sensitive indicators or Bromophenol Blue pH sensitive indicators. There may also be present in the adhesive small amounts (e.g., less than about 30% by weight, and preferably 5 to 20% by weight) of certain thermoplastic polymers such as ethylene vinyl acetate copolymers containing about 12 to 50% vinyl acetate, ethylene acrylic acid, ethylene methyl acrylate and ethylene n-butyl acrylate copolymers as well as caprolactone polymers and poly(hydroxy-butyrate/hydroxyvalerate). These polymers are employed in order to impart flexibility, toughness and strength. Alternatively, and in particular, it may be desirable to incorporate into the hot melt adhesive up to 20% by weight of certain hydrophilic polymers such as polyvinyl alcohol, hydroxyethyl cellulose, hydroxy-propylcellulose, polyvinyl methyl ether, poly(ethylene oxide), sulfonated polyester, or modified or derivatized starch which will function to increase the water sensitivity of the adhesives which may be desired for some applications.

The hot melt adhesives of the present invention may be prepared using techniques known in the art. Typically, the adhesive compositions are prepared by blending the components in the melt at a temperature of about 100 to 200°C until a homogeneous blend is obtained, approximately two hours. Various methods of blending are known and any method that produces a homogeneous blend is satisfactory. The resulting adhesives are characterized in that they have a viscosity of 50,000 cP or less at the application temperature of 350°F (177°C) or less. The viscosity as used herein is a Brookfield viscosity measured using a Brookfield viscometer model No. DV-II with spindle no. 27 at 10 rpm.

In a preferred embodiment, the hot melt adhesive of the present invention comprises:

a) 20% by weight of an SBS copolymer;

b) 57% by weight of a C_5/C_9 tackifier with a Ring and Ball softening point of 90 to 103°C;

c) 22% by weight mineral oil;

d) 0.5% by weight of an anionic surfactant;

e) 0.5% by weight polypropylene glycol; and

f) 0.5% by weight antioxidant.

The resulting adhesives of the present invention are characterized by their ability to provide a durable bond to a nonwoven or tissue article and otherwise meet the unique requirements of the application (including flexibility, non- staining, and machinable viscosity). The adhesives described herein also possess exceptional thermal stability, which distinguishes them from other moisture sensitive technologies. Further, their hydrophilic natures facilitate ready transmission of the fluid throughout the construction.

The adhesive product can be applied to a substrate such as a nonwoven article or tissue by a variety of methods including coating or spraying in an amount sufficient to cause the article to adhere to another substrate, such as tissue, nonwoven, or other conventionally employed substrates, such as polyolefin films.

As noted above, the resulting adhesives may be employed in a wide variety of uses as are known in the art. The adhesives may be effectively utilized in a variety of packaging and carton sealing applications. The non-pressure sensitive adhesives may also be used to bind a plurality of sheets in

a wide range of bookbinding operations. They may also be used for laminating tissue and/or screen-reinforced tissue layers such as are used in individual or roll use applications as in wipes, paper towels, toilet tissue and other consumer or industrial end uses. The adhesives of this invention are especially useful in the assembly or construction of various disposable articles including, but not limited to, sanitary napkins, disposable diapers, hospital gowns, bed pads and the like. In particular, adhesives are useful for the assembly of disposable articles using multi-line, spray, or slot-coating construction techniques wherein at least one flexible film substrate is bonded to at least one tissue, non-woven, polyolefin or other flexible polymeric film substrate. In addition, the adhesives may be useful in the bonding of elastic to polyethylene, polypropylene or non-woven substrate so as, for example, to impart elongation resistant gathers thereto as well as for positioning adhesives for sanitary napkins. The adhesive may also be utilized in less demanding disposable construction applications such as for end or perimeter sealing. Also, this adhesive has utility as a bandage adhesive which can be easily removed by applying water thereby preventing trauma to skin. The adhesive also finds use as a bottle labeling adhesive since the label will easily release from the bottle upon contact with water.

The following examples illustrate the compositions of suitable hot melt adhesives, and the improvement to their wicking properties as a result of the incorporation of the described surfactants.

EXAMPLES

Sample adhesives were prepared and tested for water contact angle, surface tension reduction, and rate of wicking.

The following materials were used to prepare the samples:

STEREON 841 is an styrene-butadiene-styrene ("SBS") rubber from Firestone.

MACOL DNP-10, a 10 mole ethoxylate of dinonyl phenol from PPG;

IRGANOX 1010 and 1035, hindered phenol anti-oxidants available from Ciba-Geigy;

SURFONIC DNP-100, a 10 mole ethoxylate of dinonyl phenol available from Huntsman;

HERCOLITE 290, an alpha methyl styrene oligomer from Hercules;

LUMINOL T350, a paraffinic mineral oil form Petrocanada;

VECTOR 4411, a styrene-isoprene-styrene block copolymer available from Dexco Polymers;

ECR 179EX an aromatic modified dicyclopentadiene tackifier with a 102 degree Celsius softening point available from Exxon;

FORAL NC, an ionic polar tackifier from Hercules;

PPG 3025 a polypropylene glycol with molecular weight of about 3000 available from Arco;

GENAPOL 26-L-60N, a linear alcohol ethoxylate from Clariant.

The following procedures were used to test the samples.

Contact Angle Test:

The contact angle was measured with the use of a goniometer, which has a microsyringe for dispensing accurate droplet sizes and a camera for photographing the angle of the liquid drop as it meets the surface of the solid. The contact angle is measured as the angle between the substrate and the tangent of the liquid drop (at the interface). This measurement was made 5

seconds after placing the drop on the adhesive surface. The lower the angle, the more effective the coating is in transmitting (wicking) the liquid through the discontinuous adhesive layer.

Viscosity:

Viscosity measurements were determined after 30 minutes using a Brookfield viscometer (Spindle 27).

Heat Stability:

Some of the adhesives were also tested for thermal stability by storing at 275°F for 24 hours. After the testing period, remove the jar, check for the following:

a. Skin

b. Dirt/char particles

c. Sedimentation - partial skin precipitating and falling to the bottom of the jar.

d. Volatile

e. Gelation - carefully examine the contents with a glass stirring rod for signs of gels or lumps.

f. Color or odor

g. Product separation - the presence of distinct layers, also known as phasing.

In the following tables, "pass" indicated that there was no evidence of skin, gel, separation or sedimentation.

Tensile Strength:

The adhesive was heated to 149° to 177°C in a glass jar. Molten adhesive was poured into a silicone rubber mold which was one inch square at

each end and one half inch square in the gage section and 1/8 inch thick. At least three samples were prepared for each product. These were conditioned overnight at 70°F and 50% relative humidity ("RH"). Tensile specimens were pulled on an Instron at 12 inches/minute.

Surface Tension Reduction (STR)

The water surface tension was measured using the Dunuoy ring method. Two grams of adhesive were placed in a clean 110 ml glass jar with a 5 cm inner diameter, melted in a 135°C oven, and then cooled to room temperature. Twenty ml of saline solution (0.85% NaCl in deionized water) was then added to this jar. The surface tensions of a pure saline solution and of the saline solution after 15 minutes exposure to the adhesive were measured using a KRUS K-14 tensionmeter. The difference in the surface tensions were recorded as the surface tension reduction, ("STR").

EXAMPLE I

Sample 1-A is an adhesive prepared according to the present invention comprising an anionic surfactant, AEROSOL OT. Sample 1-B is an adhesive prepared according to the present invention comprising an anionic surfactant, AEROSOL OT and PPG 3025. Sample 1-D is a comparative example prepared according to U.S. Patent No. 5,532,306.

Table 1

	1-A	1-B	1-C	1-D
STEREON 841A	20	20	20	-
VECTOR 4411	-	-	-	28
ECR 179EX	57	57	57	-
FORAL NC	-	-	-	56
LUMINOL T-350	22	21	22	-
AEROSOL OT-100	0.5	2	1.0	-
PPG-3125	0.5	-	-	-
MACOL DNP-10	-	-	-	16
IRGANOX 1010	0.2	0.2	0.2	0.5
IRGANOX 1035	0.5	0.5	0.5	-
Contact angle	34.5°	37.5°	53°	66°
STR (dynes/cm)	4.1	16.7	-	19.0

The results show that Samples 1-A, 1-B and 1-C, have a water contact angle which is lower than the adhesive prepared according to U.S. Patent No. 5,532,306. In addition, Sample 1-D comprises FORAL NC, a polar tackifier necessary to ensure compatibility with the higher level of surfactant used.

EXAMPLE 2

In this example, adhesive formulations, Samples 2-A and 2-B, with GENAPOL 26-L-60N, a nonionic surfactant, were prepared and the physical properties measured. Sample 2-C is a conventional rubber based adhesive.

TABLE 2

	2-A	2-B	2-C
STEREON 841A	20	20	20
ECR 179EX	53	53	57
HERCOLITE 290	4	4	-
LUMINOL T-350	15	17	23
GENAPOL 26-L-60N	8	6	-
IRGANOX 1010	0.2	0.2	0.2
IRGANOX 1035	0.5	0.5	0.5
Viscosity @ 275°F	3,900	3,000	4,300
Heat Stability: 24 hr. @350°F	Pass	Pass	Pass
Water contact angle			
@ 5 seconds	41	32°	98.5°
@ 30 seconds	<30°	<30°	98.5°

These results indicate that a low water contact angle can be achieved with low levels of a linear nonionic surfactant, GENAPOL, when compared to Sample 1-D of Example 1.

EXAMPLE 3

The following formulations were prepared with PPG, but without surfactant. Sample 3-C is the same conventional rubber based adhesive used in Sample 2-C.

	3-A	**3-B**	**3-C**
STEREON	20	20	20
ECR 179EX	57	57	57
LUMINOL T350	21	19	23
PPG 3025	2.0	4.0	0
IRGANOX 1010	0.2	0.2	0.2
IRGANOX 1035	0.5	0.5	0.5
STR	6.8	8.5	5.5
Contact Angle	49.6	44.6	96°

The results indicate how low levels of PPG give very low STR and low contact angle when added to the control formulation in appropriate amounts.

EXAMPLE 4

A hot melt adhesive was prepared with AEROSOL OT-100, an anionic surfactant, and PPG. In Table 4, Sample 1-A was prepared according to Example 1.

TABLE 4

	4-A	1-A	4-C
STEREON 841-A ECR 179EX LUMINOL T-350 AEROSOL OT-100 PPG-3025 IRGANOX 1010 IRGANOX 1035	20 57 21 1.0 1.0 0.2 0.5	20 57 22 0.5 0.5 0.2 0.5	20 57 21.5 1.0 0.5 0.2 0.5
Viscosity @ 275°F	4150 cps	4438 cps	4175 cps
Heat Stability 24 hrs. @ 350°F	Pass	Pass	Pass
Water Contact Angle 75 hrs. @ RT @ 5 sec.	39°	34°	33°

The results shown above in Table 3 indicate that low levels of the anionic surfactant Aerosol OT-100 when combined with PPG-3025 give a lower water contact angle. Sample 4A which comprises both PPG and surfactant, provides a lower contact angle than Sample 3-A from Example 3 which only comprises PPG or Sample 1-C from Example 1 which only comprises surfactant.

EXAMPLE 5

Comparative of an adhesive prepared with an anionic and a nonionic surfactant.

TABLE 5

	5-A	1-A	5-C
STEREON	20	20	20
ECR 179EX	53	57	53
HERCOLITE 290	4.0	--	4.0
GENAPOL-26-L-60N	6.0	--	8.0
AEROSOL OT-100	--	0.5	--
PPG 3025	--	0.5	--
LUMINOL T350	17	22	15.0
IRGANOX 1010	0.2	0.2	0.2
IRGANOX 1035	0.5	0.5	0.5
End Seal Strength g/in			
Dry	201	145	
Wet	86	147	

End seal strength was measured by bonding polyethylene film to polypropylene spun bond nonwoven on a coater machine at a speed of 380 feet/min using a 3 spiral spray application head which gives a total coverage width of 2.25 inches at a temperature of 265 –275°F, and 2.66 gram/meter^2 add on level.

Bond strength was measured by pulling bonds at 10 inch/min. along the machine direction on an Instron testing machine. Dry bonds were measured after conditioning for 24 hours at 50% RH and 70°F. Wet bonds were conditioned as above then immersed in water for one hour and tested immediately thereafter.

The results are reported above in Table 5.

The bond strength of Sample 5-A, comprising nonionic surfactant, is lowered after one hour soak in water. Sample 1-A, comprising anionic

surfactant with PPG, showed no decrease in end seal value, indicating good wet bond strength. These results show the advantages of using an anionic surfactant versus a nonionic surfactant. These advantages include a low water contact angle, with a much lower level of anionic surfactant versus a nonionic surfactant.

EXAMPLE 6

An anionic surfactant and PPG were added to a conventional leg elastic adhesive, Sample 6-A. Sample 6-B is the same conventional rubber based adhesive used to prepare Sample 6-A. The viscosity at 275°F was 21,575 cps; water contact angle at 5 seconds was 53°. Tensile strength was measured at 12"/min. and the following data obtained:

TABLE 6

	6-A	6-B
34-5620	99	100
AEROSOL OT-100	0.5	-
PPG-3025	0.5	-
Viscosity (cps)		
@275°F	27,700	31,600
@300°F	12,600	15,300
@325°F	7,000	8,300
@350°F	4,200	4,600
Tensile (psi)		
Yield	17	16
Ultimate	234	193
Break stress	226	175
% strain @ break	1,469	1,476
Stress @ 500%	43	31
Contact angle	53°	98.5°

These results show that a low water contact angle can be achieved with low levels of PPG-3025 and Aerosol OT-100, with minimal impact on the physical characteristics of the adhesive.

EXAMPLE 7

This example evaluated the recyclability of adhesives used for labeling PET bottles. With the increasing demands being placed upon packaging manufacturers to ensure their products can be recycled or reused, bottle washing efficiency is becoming more important. Bottle labelers are demanding adhesives which are sensitive to water enabling good label release or adhesive removal from the PET bottle.

The adhesive sample was heated to application temperature, 130°C. A thin coating of adhesive was applied to a 3cm by 8cm piece of label stock by pulling a K-bar over a bead of hot adhesive, drawing the adhesive down over the label stock to apply a thin coating of adhesive. The coated label stock was immediately bonded to PET. The adhesive was reactivated by heating the bond on a hot plate for a few seconds to ensure an efficient bond is made. The bond was then immersed in a 3 liter neutral water bath at 50°C with agitation at a speed of 850 rpm to mimic the conditions found in typical bottle washers. The time it takes for the label to separate from the PET and how much adhesive remains on the PET is noted.

The following adhesive formulation was tested:

	7-A
VECTOR 4411	16
FORAL NC	60
GENAPOL 26-L-60	7
KAYDOL Mineral Oil	17
IRGANOX 1010	0.5
Viscosity (cps)	
@225°F	7,500
@250°F	1,500
@275°F	600

The results show that polypropylene label stock bonded to PET with Sample 7-A takes 55±15 minutes to separate. The adhesive remains on the polypropylene stock and releases from the PET. This is preferable since adhesive that can not be fully removed causes various small particles, such as plastics, dust, paper fibers and sand, to adhere to the adhesive residues on the PET surface, contaminating the regenerated PET flakes.

The control was a commercial grade bottle labeling adhesive, ETM 130E, which showed negligible water sensitivity; the label stayed on the bottle under the above conditions.

What is claimed:

1. A hot melt adhesive composition comprising:

(a) 5 to 40% by weight, of an A-B-A linear or radial rubbery block copolymer;

(b) 20 to 70% by weight of a compatible tackifying resin;

(c) 0 to 30% plasticizer;

(d) 0 to 2% stabilizer; and

(e) one or more of the following:

(i) 0.1 to 20% of one or more polyethers;

(ii) 0.1 to 9% of a surfactant; or

(iii) a combination of (i) and (ii).

the adhesive composition exhibiting a contact angle of 90° or less and causing a reduction in fluid surface tension of less than about 40 dynes/cm.

2. A hot melt adhesive according to Claim 1 wherein the A-B-A block copolymer is a styrene-butadiene-styrene block copolymer comprising at least 35% styrene.

3. A hot melt adhesive composition comprising:

(a) 5 to 40% by weight, of an A-B-A linear or radial rubbery block copolymer;

(b) 20 to 70% by weight of a compatible tackifying resin;

(c) 0 to 30% plasticizer;

(d) 0 to 2% stabilizer;

(e) 0.1 to 20% of one or more polyethers; and

(f) 0.1 to 9% of a surfactant,

the adhesive composition exhibiting a contact angle of 90° or less and causing a reduction in fluid surface tension of less than about 40 dynes/cm.

4. A hot melt adhesive composition comprising:

(a) 5 to 40% by weight, of an A-B-A linear or radial rubbery block copolymer;

(b) 20 to 70% by weight of a compatible tackifying resin;

(c) 0 to 30% plasticizer;

(d) 0 to 2% stabilizer; and

(e) 0.1 to 9% of a surfactant, the adhesive composition exhibiting a contact angle of 90° or less and a reduction in surface tension of less than about 40 dynes/cm.

5. A hot melt adhesive composition comprising:

(a) 5 to 40% by weight, of an A-B-A linear or radial rubbery block copolymer;

(b) 20 to 70% by weight of a compatible tackifying resin;

(c) 0 to 30% plasticizer;

(d) 0 to 2% stabilizer; and

(e) 0.1 to 20% of one or more polyethers; the adhesive composition exhibiting a contact angle of 90° or less and a reduction in surface tension of less than about 40 dynes/cm.

6. A disposable article comprising a hot melt adhesive according to Claims 1, 2, 3, 4 or 5.

7. A bottle label applied with a hot melt adhesive according to Claims 1, 2, 3, 4 or 5.

INTERNATIONAL SEARCH REPORT

Inter   nal Application No
PCT/US 00/16943

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 C09J153/02 C08L53/02 A61L15/58

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 7 C09J C08L A61L

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, WPI Data, PAJ

C. DOCUMENTS CONSIDERED TO BE RELEVANT

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X	EP 0 699 727 A (NAT STARCH CHEM INVEST) 6 March 1996 (1996-03-06) cited in the application * abstract ; page 2, line 34-56 ; page 3, line 4-26 ; page 3, line 48-51 ; page 6, line 9-11 ; Table 1 ; page 4, line 20-31 ; claims 1-9 ; page 4, line 7-12 * page 3, line 58 -page 3, line 59 ---	1-7
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[X] Further documents are listed in the continuation of box C.

[X] Patent family members are listed in annex.

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Date of the actual completion of the international search	Date of mailing of the international search report
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INTERNATIONAL SEARCH REPORT

Inter   nal Application No
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